NATO UNCLASSIFIED

NORTH ATLANTIC TREATY ORGANIZATION
ORGANISATION DU TRAITE DE L'ATLANTIQUE NORD

MILITARY AGENCY FOR STANDARDIZATION (MAS)
BUREAU MILITAIRE DE STANDARDISATION (BMS)
1110 BRUSSELS

MAS/32-MMS/4300
29 January 1993

To : See MAS Distribution List No. 2

Subject : STANAG 4300 MMS (EDITION 1) - TEST PROCEDURES FOR ASSESSING THE QUALITY OF ALUMINIUM POWDER, FOR USE IN EXPLOSIVE FORMULATION, FOR DELIVERIES FROM ONE NATO NATION TO ANOTHER

Reference : AC/310-D/84 dated 23 June 1989

Enclosure : STANAG 4300 (Edition 1)

1. The enclosed NATO Standardization Agreement which has been ratified by nations as reflected in page iii is promulgated herewith.

2. The reference listed above is to be destroyed in accordance with local document destruction procedures.

3. AAP-4 should be amended to reflect the latest status of the STANAG.

ACTION BY NATIONAL STAFFS

4. National staffs are requested to examine page iii of the STANAG and if they have not already done so, to advise the Defence Support Division, IS, through their national delegation as appropriate of their intention regarding its ratification and implementation.

E. STAI
Major-General, NOAF
Chairman, MAS

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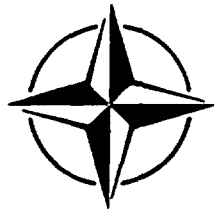
MAS32A

STANAG 4300
(Edition 1)

NORTH ATLANTIC TREATY ORGANIZATION

(N A T O)



MILITARY AGENCY FOR STANDARDIZATION

(M A S)

STANDARDIZATION AGREEMENT

SUBJECT : TEST PROCEDURES FOR ASSESSING THE QUALITY OF ALUMINIUM POWDER, FOR USE IN EXPLOSIVE FORMULATION, FOR DELIVERIES FROM ONE NATO NATION TO ANOTHER

Promulgated on 29 January 1993

E. STAI
Major-General, NOAF
Chairman, MAS

STANAG 4300
(Edition 1)

RECORD OF AMENDMENTS

No.	Reference/date of amendment	Date entered	Signature

EXPLANATORY NOTES

AGREEMENT

1. This NATO Standardization Agreement (STANAG) is promulgated by the Chairman MAS under the authority vested in him by the NATO Military Committee.

2. No departure may be made from the agreement without consultation with the tasking authority. Nations may propose changes at any time to the tasking authority where they will be processed in the same manner as the original agreement.

3. Ratifying nations have agreed that national orders, manuals and instructions implementing this STANAG will include a reference to the STANAG number for purposes of identification.

DEFINITIONS

4. Ratification is "The declaration by which a nation formally accepts the content of this Standardization Agreement".

5. Implementation is "The fulfilment by a nation of its obligations under this Standardization Agreement".

6. Reservation is "The stated qualification by a nation which describes that part of this Standardization Agreement which it cannot implement or can implement only with limitations".

RATIFICATION, IMPLEMENTATION AND RESERVATIONS

7. Page iii gives the details of ratification and implementation of this agreement. If no details are shown it signifies that the nation has not yet notified the tasking authority of its intentions. Page iv (and subsequent) gives details of reservations and proprietary rights that have been stated.

Agreed English/French Texts

STANAG 4300
(Edition 1)

NAVY/ARMY/AIR

NATO STANDARDIZATION AGREEMENT
(STANAG)

TEST PROCEDURES FOR ASSESSING THE QUALITY OF ALUMINUM POWDER, FOR USE IN EXPLOSIVE FORMULATION, FOR DELIVERIES FROM ONE NATO NATION TO ANOTHER

Related documents:

British Standard Institute BS 4359, Part 2, 1982
International Standard ISO 3923/1
International Standard ISO 3923/2
International Standard ISO 3953
ASTM E-101-67
ASTM E-34

AIM

1. The aim of this agreement is to standardize methods of test for assessing the quality of aluminum powder used in the manufacture of explosives, propellants and pyrotechnics. The use of these methods should facilitate cross procurement and provide means by which countries can satisfy themselves that aluminum received from abroad has been tested by acceptable means. They are not intended for use as quality control purposes during the manufacture of aluminum powder.

AGREEMENT

2. Participating nations agree to use the test procedures described for assessing the quality of aluminum powder when required by the procuring nation.

Part I - Aluminum Covered and List of Tests

3. Description of Aluminum - The aluminum powder used in the manufacture of high explosives, propellants and pyrotechnics for military purposes is prepared by atomisation, stamping or grinding of aluminum metal. Atomisation produces a spheroidal or elongated particle, stamping yields a particle described as "flake" and grinding yields irregularly shaped, elongated particles.

4. Tests to be Applied - The tests to be applied will depend upon the type and the specification of the aluminum powder being procured. The end use of the material determines the specification of its physical and chemical properties. Because of the great variety of aluminum powders available, this document does not seek to identify various grades and types. Specifications for properties such as particle shape, size and size distribution, chemical purity, etc. shall be made in the purchaser's order.

4.1 List of Physical Tests

(1) Visual Examination
(2) Sieve Test
(3) Determination of Average Particle Size
(4) Determination of Particle Size Distribution
(5) Determination of Apparent Density
(6) Determination of Tap Density

4.2 List of Chemical Tests

(1) Determination of Volatile Matter
(2) Determination of Matter Soluble in diethyl ether
(3) Determination of pH of Aqueous Extract
(4) Determination of Matter Insoluble in Hydrochloric Acid
(5) Determination of Grit
(6) Determination of Free Metallic Aluminum
(7) Determination of Elemental Impurities

Part II - Description of Physical Tests

5. Visual Examination - A sample of the aluminum powder will be examined under a microscope of suitable magnification to permit clear viewing of individual particles. Atomized particles shall have an approximately spherical shape. Flake particles shall consist of irregular, flattened particles with frayed, irregular contours. Ground particles shall consist of irregular, elongated particles.

6. Sieve Test - The sieve test shall be conducted using a Tyler Ro-Tap Testing Sieve Shaker and US Standard sieves (RR-S-366), or equivalent. The stack requires sieves with the coarsest sieve on top and the finest sieves on the bottom. Place a catch pan at the bottom of the stack. Weigh to the nearest 0.02 g a 50 g sample and brush onto the uppermost sieve. Place a sieve cover on top of the stack and lock stack on the sieve shaker. Shake for 15 minutes. Carefully brush material passing the bottom sieve onto a weighing pan and weigh to the nearest 0.01 g. Add material from the next higher sieve and weigh. Continue until all the material has been weighed. Multiply each

weight by 2 to obtain weight cumulative percent passing at each sieve level. Repeat analysis if total recovered weight is less than 99 percent. Sieving flake aluminum can sometimes cause problems. In such cases, wet the sample with white spirit, and wash the material through the sieve using a jet of this liquid. Wash the material retained in the sieve with acetone, dry and weigh.

7. Determination of Average Particle Size - The average particle size shall be determined using the Fisher Sub-Sieve Sizer procedure described hereafter. An acceptable alternate procedure is described in BS 4359, Part 2 dated 1982.

7.1 Checking of Apparatus - Check water level in standpipe of pressure regulator. Adjust water level as required. Check drying agent in rear of cabinet. Remove and dry in oven if indicator is pink. Check sample packing assembly to see that the pointer tip coincides with the base line on the calculator chart. Check calibration with Fisher Sub-Sieve Sizer Calibrator. If calibration is off, adjust flowmeter wires according to calibration instruction.

7.2 Procedure - Attach porous plug to plug manipulator. Center a filter paper disc over end of sample tube and push plug into sample tube with perforated surface against filter paper disc. Remove plug manipulator and place sample tube in vertical position on metal post of support stand to force plug to proper height inside sample tube. Weigh to nearest 0.01 g a sample of powder equal in grams to the true density of sample (2.7 g). With a funnel, transfer weighed sample to sample tube. Tap side of tube to settle powder. Center another filter paper disc over the top of the tube and force a second plug (perforated surface against paper disc) into tube. Push plug and disc downward until powder is compacted against lower plug and disc. Place sample tube on post under rack and pinion with lower plug in contact, with post. Lower rack until upper plug is in contact with bottom of the rack. Turn pinion knob by applying standard torques until sample is packed to optimum porosity. Shift Calculator Chart laterally until tip of pointer coincides with the Sample Height Curve. Place sample tube (without disturbing sample) between rubber cushion supports. Clamp upper tube down until air-tight seal is obtained at both ends. Turn on switch. When liquid level in manometer has reached maximum height turn pinion knob until bar on rack is at bottom of meniscus in manometer. Read particle size at tip of pointer. Fine powders may require five minutes or more running time before maximum height is reached. If the average particle size (APS) is 0.2 to 20 microns, read chart directly. If APS is between 20 and 50 microns, turn range control indicator to left and double chart readings.

8. Determination of Particle Size Distribution - The size distribution of spheroidal particles shall be determined using Malvern Particle Sizers 2600 or 3600 mounted with a suitable focal lens and a PSl small volume (15 cm^3) non-recirculating cell. Other brands of particle size analysers such as Microtrac based on the phenomenon of low angle forward-scattering light from a laser beam through a cloud of particles suspended in liquid will be acceptable.

8.1 Procedure - Install the non-recirculating cell against the collecting lens of the Malvern and fill it with isopropanol. Prepare a light dispersion of aluminum powder (2-5 g) in mechanically stirred isopropanol (about 50 cm^3) contained in a 150 cm^3 beaker sitting in an ultrasonic bath. Do a background reading of the cell. Using a dropper, rapidly add a few drops of the dispersion into the cell until the obscuration is between 0.1 to 0.4 and then do the measurement. Selecting the model independant analysis package, determine particle size distribution and average particle size on a volume basis.

9. Determination of Apparent Density - The apparent density of powder that flows freely through a 5 mm orifice shall be determined in accordance with ISO 3923/1 titled 'Metallic Powders - Determination of Apparent Density Part 1: Funnel Method'. The apparent density of powder that does not flow freely through a 5 mm orifice shall be determined in accordance with ISO 3923/2 - titled 'Metallic Powders - Determination of Apparent Density - Part 2: Scott Volumeter Method'.

10. Determination of Tap Density - The tap density shall be determined using calibrated rubber-stoppered measuring cylinders 25 cm^3 in capacity and length approximately 15 cm, and 100 cm^3 in capacity and length approximately 25 cm. The upper surface of a wooden base stand is covered with hard leather. A guide is provided by placing two wooden filter rings, clamped one above the other, on the same support. The lower ring is positioned to limit the travel of the cylinder to 6.5 cm. The tap density determined in accordance with ISO 3953 titled 'Metallic Powders - Determination of Tap Density' is an equivalent acceptable technique.

10.1 Procedure (blown grades) - Transfer 20 ± 0.1 g of the sample to the 25 cm^3 cylinder and close with the rubber stopper. Place the cylinder into the wooden stand and drop it vertically 30 times from a height of 6.5 cm on to the leather pad. Level off the surface of the powder by the minimum amount of side-tapping and read off the volume (V_1) occupied by the powder.

10.2 Procedure (flake grades) - Transfer 15 ± 0.1 g of the sample to the 100 cm^3 cylinder, and add 60 cm^3 of methylated spirit 92% v/v. Stopper cylinder and shake for about 5 minutes. Wash down any material adhering to the walls of the cylinder with a jet of methylated spirit and allow cylinder to stand in a vertical position for one hour. Read off volume (V_2) occupied by the aluminum powder.

Calculation

Apparent density (blown grades) $g/cm^3 = \frac{20}{V_1}$

Apparent density (flake grades) $g/cm^3 = \frac{15}{V_2}$

Part III - Description of Chemical Tests

11. Determination of Volatile Matter - Heat a weighing bottle in a drying oven at about 105°C for 2 hours. Cool in a desiccator for 30 minutes with cover off. Weigh bottle and cover to the nearest 0.1 mg. Record as W1. Add approximately 5 g of the sample to weighing bottle and weigh to nearest 0.1 mg with cover in place. Record as W2. Remove cover and place bottle and cover in drying oven or preferably in dry nitrogen stream at 105°C for 3 hours. Remove bottle and cover from oven and allow to cool in desiccator for 30 minutes. Place cover on bottle and reweigh to nearest 0.1 mg. Record as W3. Calculate as follows:

$$\text{Percent Volatiles} = \frac{(W2-W3) \times 100}{W2-W1}$$

12. Determination of Matter Soluble in diethyl ether - Dry a clean 400 cm^3 flask in an oven at 90°C, and cool in a desiccator to constant weight. Weigh to nearest 0.1 mg and record as W1. Weigh about 10 g of the sample to the nearest 0.1 mg and extract for 4 hours with 100 cm^3 of diethyl ether using a Soxhlet extraction apparatus with a Whatman single thickness, fat free extraction thimble or equivalent. After extraction has been completed, evaporate the diethyl ether almost to dryness using a boiling water bath. Dry residue in an oven at 90°C to a constant weight and then cool in a desiccator. Weigh and record this as W2. Run a blank with each group of the tests and record this as W3. Calculate the following quantity:

$$\text{Percent Matter Soluble} = \frac{((W2-W1)-W3) \times 100}{\text{Weight of sample}}$$

Diethyl ether is flammable, and appropriate safety precautions against accidental ignition must be taken. Furthermore peroxides contained in ether can constitute an explosive hazard.

13. Determination of pH of Aqueous Extract - The determination of pH shall be done using any standard pH meter accurate to 0.05 pH unit and calibrated using suitable buffer solutions. Transfer 5 ± 0.1 g of the sample to a 250 cm^3 beaker, add 50 cm^3 of water and stir for 2 minutes. Allow powder to settle for about 1 minute and determine the pH of the aqueous extract.

14. Determination of Matter Insoluble in Hydrochloric Acid - Transfer 10 ± 0.1 g of the sample to a 600 cm^3 beaker and add 50 cm^3 of water. Add hydrochloric acid (d = 1.18 g cm^{-3}) dropwise, controlling the reaction by the rate of addition of the acid, until the sample dissolves. Heat the beaker gently for 10 minutes. Dilute with water, allow any solid matter to settle and decant the clear liquid. Wash the residue with water and repeat the decantation. Transfer the residue to a 400 cm^3 beaker, add 20 cm^3 of 50 % sodium hydroxide solution and boil gently for 10 minutes. Dilute the solution immediately with 200 cm^3 of water, allow the residue to settle and carefully decant the supernatant liquid. Repeat the washing twice with water and finally with 2 % hydrochloric acid solution. Filter any residue in the beaker through a No. 40 Whatman filter-paper or equivalent and wash well with hot water. Dry the filter-paper and ignite it in a tared (W1) ignited porcelain crucible at a temperature of 800°C for 30 minutes. Cool in a desiccator for 30 minutes and reweigh (W2). Retain the residue and crucible for the determination of grit.

$$\text{Total insoluble matter (\%)} = (W2 - W1) \times 10$$

15. Determination of Grit - Cautiously mix one volume of nitric acid, 1.42 g/cm^3 with three volumes of hydrochloric acid, 1.18 g/cm^3. Transfer the residue from clause 14 to a 100 cm^3 beaker add 5 cm^3 of aqua regia and boil gently for 5 minutes. Cool, dilute the contents of the beaker with water and neutralize with sodium hydroxide solution. Pour the liquid through a 63 µm sieve or equivalent ensuring all the residue is transferred to the sieve. Wash the residue thoroughly with water and dry the sieve in an oven at 103 ± 2°C. Brush gently the material retained on the sieve with a small soft-hair brush. Transfer any residue retained on the sieve to a tared watch glass (W1) and weigh (W2). Transfer the residue on to the 250 µm sieve or an equivalent and again brush gently with a soft-hair brush. Transfer any residue retained to a tared watch glass (W3) and weigh (W4).

Grit retained on a 63 µm sieve, (%) = (W2 - W1) X 10.
Grit retained on a 250 µm sieve, (%) = (W4 - W3) X 10.

16. Determination of Free Metallic Aluminum - The free metallic aluminum shall be determined by measuring the volume of gas evolved when a weighed quantity of aluminum powder is reacted with sodium hydroxide solution

$$2Al + 2NaOH + 2H_2O = 2\ NaAlO_2 + 3H_2\uparrow$$

A method equivalent to the procedure described hereafter will be acceptable.

16.1 Apparatus - The basic apparatus is depicted in Figure 1. It is advisable to house the apparatus in a separate room, free from draughts and away from direct sunlight and sources of heat.

16.2 Reagents - 20 % M/V sodium hydroxide solution.

16.3 Calibration of receiver - Liquids may be introduced into (R) by attaching a tube to the outlet of tap S2 which dips into a vessel containing the required liquid, turning S1 and S2 to the appropriate positions and applying suction to the open arm of S1. Thoroughly clean the receiver (R) with chromosulfuric (chromic) acid and rinse well with water. Fill the bottle (B) about three-quarters full with water. Ensure that the open arm of S2 is free from liquid by applying gentle pressure to the open arm of S1 to blow out any trapped liquid. Turn S2 to connect (R) with (B) and apply gentle pressure (using manometer (M)as guide) via tap S3 to force water into (R) until it is filled to the mark A. Check that no air bubbles are trapped on the walls of (R). Close tap S2 and release the pressure on S3. Open tap S2 to the exit arm and run out the volume of water, contained in (R), into a suitable tared receiver (W1 ± 10 mg) and weigh (W2 ± 10 mg). Note the temperature of the water (T_1°C).

$$\text{Volume of receiver } (V_1) = \frac{W2-W1}{d_1}$$

where d_1 = density of water at T_1°C

16.4 Procedure - Ensure that the bottle (B) is at least three-quarters full of water. Open tap S1 to the atmosphere and adjust tap S2 to connect (B) with (R). Open tap S3 and apply gentle pressure (using manometer (M) as guide) via the open limb of S3 to force water into (R) until it is filled to just above the mark A. Close tap S2 and release the pressure on tap S3. Carefully adjust tap S2 to allow the level of water in (R) to fall to coincide with the mark A. Adjust tap S1 so that both (R) and (X) are connected to the atmosphere. Fill the burette with sodium hydroxide solution and adjust the level to zero and wash any excess alkali from the tip of the burette to prevent any premature reaction with the sample. Weigh accurately 0.355-0.365 g (W3) of the sample and transfer quantitatively to the reaction vessel (X). Add 20 cm^3 of water to (X) and immediately connect it firmly to the apparatus. Allow (X) and its contents to reach equilibrium and, after ensuring that the level of water in (R) is still at the mark A, adjust S1 so that (R) is connected to (X) only. Close tap S3 and adjust tap S2 to connect (R) with (B). Record the temperature (T_2) shown by thermometer (C). Check the position of all taps and run the sodium hydroxide dropwise into (X), controlling the rate of addition of alkali very carefully to prevent gas being evolved too quickly. If necessary the reaction flask may be cooled by immersing it in a beaker of cold water. Maintain the pressure in (R), as indicated by the manometer (M), slightly below atmospheric by opening tap S3 from time to time during the evolution of hydrogen. Continue the addition of

alkali until a total of 20.0 cm^3 has been added to (X). Swirl the contents of (X) to ensure that no unreacted sample remains. Allow the apparatus to stand for approximately 1 hour, to allow the temperature to reach equilibrium. Note the temperatures (T_3)°C and (T_4)°C indicated by the thermometers (C) and (D) respectively. Adjust the level of the water in the receiver (R) to that of the water in the manometer (M) by releasing or applying pressure to the open end of S3. Close tap S2. Owing to the capillary effects, the liquid levels in (R) and (M) are in equilibrium when the bottom of the meniscus in (M) is level with the top of the meniscus in (R). Turn S1 to open (R) to the atmosphere and run the remaining water from (R) through S_2, into a tared 250 cm^3 conical flask (W4 ± 10 mg). Weigh the flask immediately (W5 ± 10 mg). Record the atmospheric pressure (P).

16.5 Calculation - Volume of water remaining in receiver R.

$$V_3 = \frac{W5 - W4}{d_2}$$

where d_2 = density of water at temperature T_4°C.

Free metallic aluminium (uncorrected) ... per cent =

$$\frac{(P-x-y) \times (V_1-V_3-V_2) \times 0.02884}{(273.15 + T_4) \times W3} \quad \ldots\ldots \text{A}$$

where P = barometric pressure (mm)
x = water vapour pressure at temperature T_3 (mm)
y = barometric correction (for brass scale) (mm)
V_1 = volume of receiver (R) cm^3
V_2 = volume of sodium hydroxide solution added (cm^3)
V_3 = volume of water remaining in the receiver (cm^3)
T_4 = ambient temperature of apparatus (°C)
W3 = weight of sample (g)

When quantities of elemental zinc and silicon are present in the sample the following correction must be made:

Free metallic aluminium (corrected) ... (%) =

A - (% zinc content x 0.27) - (% silicon content x 1.28).

NOTE: It is advisable to try to ensure that the final temperature T_3 is within 0.2°C of the initial temperature T_2 otherwise the following correction will be significant and must be taken into consideration.

16.6 Dead space correction - Volume of hydrogen evolved will be:

$$(V_1 - V_2 - V_3) + C$$

where C is the volume correction to be applied due to the expansion or contraction of gas in the dead space.
If T_2 differs from T_3 then:

$$C = \frac{\text{(volume of dead space)} \times (T_2 - T_3)}{273.15}$$

if T_3 is greater than T_2 then 'C' is negative.

17. Determination of Elemental Impurities - The percent of Elemental Impurities such as silicon, copper, iron, zinc, magnesium, lead, chromium, etc. shall be determined using the 'Spectrographic Analysis of Aluminum and Aluminum Alloys by Point-to-Plane Technique' in accordance with ASTM E-101-67 or an equivalent procedure acceptable to the national procurement authority. Alternate techniques to determine the Elemental Impurities will be the Atomic Absorption Spectroscopy or chemical analysis in accordance with ASTM E 34 (Chemical Analysis of Al and Al Based Alloys) or an equivalent procedure.

18. Deviations - Deviations from Standard Procedures specified on the purchase order or requested by the manufacturer shall be reported to the buying nation.

IMPLEMENTATION OF THE AGREEMENT

19. This STANAG is implemented when a nation has issued the necessary orders/instructions putting the contents of this agreement into effect.

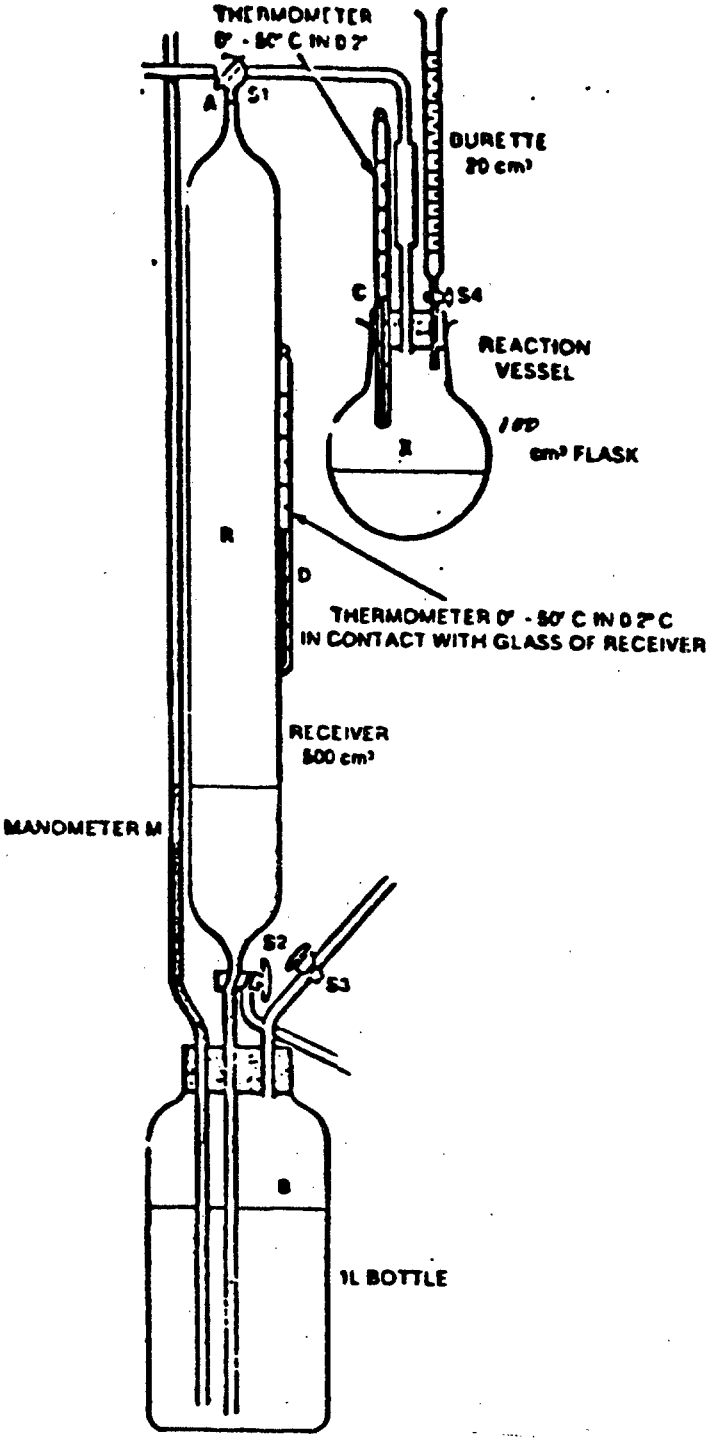


FIGURE 1 - Hydrogen Evolution Apparatus
for the Determination of Free Metal

RATIFICATION AND IMPLEMENTATION DETAILS
STADE DE RATIFICATION ET DE MISE EN APPLICATION

NATION	NATIONAL RATIFICATION REFERENCE DE LA RATIFICATION NATIONALE	NATIONAL IMPLEMENTING DOCUMENT NATIONAL DE MISE EN APPLICATION	IMPLEMENTATION/MISE EN APPLICATION					
			FORECAST DATE DATE PREVUE			ACTUAL DATE DATE REELLE		
			NAVY MER	ARMY TERRE	AIR	NAVY MER	ARMY TERRE	AIR
BE								
CA								
DA	M.204.69-S 4300/ML4-21329 of/du 3.10.89	STANAG				4.93		4.93
FR								
GE	BMVg Fü S IV 1 Az 03-51-60/4300 of/du 22.2.90		5.93	5.93	5.93			
GR								
IT								
LU								
NL	M90/0198/0513 of/du 23.1.90					1.93	1.93	1.93
NO	MAS-324/90/B/HST/U3/B0/KAS/STANAG 4300 of/du 8.3.90	STANAG 4300				4.93	4.93	4.93
PO								
SP								
TU								
UK	D/D Stan/341/8/4300 pf/du 10.8.90	STANAG				4.93	4.93	4.93
US	AMCICP-AA of/du 13.1.92	MIL-A-512				1.93	1.93	1.93